



Applicant's Docket No. U 013394-2

SS

PATENT

GAU 2652  
#4

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Solomon Sundar MANOHARAN  
Serial No.: 09/829,414  
Filed: April 9, 2001  
For: A MAGNETO-RESISTIVE CrO<sub>2</sub> POLYMER COMPOSITE BLEND

Group No.: 2652  
Examiner:

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Assistant Commissioner for Patents  
Washington, D.C. 20231

Technology Center 2600

TRANSMITTAL OF CERTIFIED COPY

Attached please find the certified copy of the foreign application from which priority is claimed for this case:

Country: India  
Application Number: 934/DEL/2000  
Filing Date: November 13, 2000

WARNING: "When a document that is required by statute to be certified must be filed, a copy, including a photocopy or facsimile transmission of the certification is not acceptable." 37 C.F.R. 1.4(f) (emphasis added).

CERTIFICATE OF MAILING (37 C.F.R. 1.8a)

I hereby certify that this correspondence is, on the date shown below, being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

Date: July 25, 2001

Signature

JOHN RICHARDS

(type or print name of person certifying)

(Transmittal of Certified Copy—page 1 of 2) 5-4

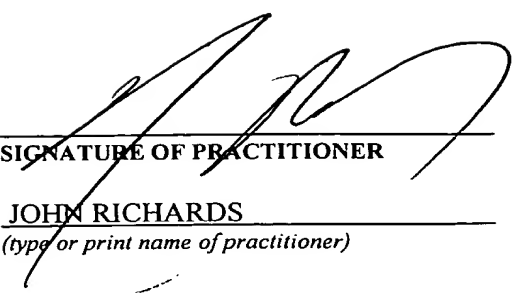
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NOTE: "The claim to priority need be in no special form and may be made by the attorney or agent, if the foreign application is referred to in the oath or declaration, as required by § 1.63." 37 C.F.R. 1.55(a).

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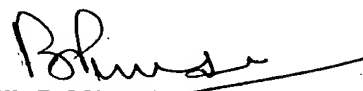


GOVERNMENT OF INDIA  
PATENT OFFICE BRANCH,  
MUNICIPAL MARKET BUILDING,  
IIIIRD FLOOR, KAROL BAGH,  
NEW DELHI-110 005.

#4  
1-12-02

I the undersigned being an officer duly authorized in accordance with the provision of the Patent Act, 1970 hereby certify that annexed hereto is the true copy of the Application, Complete Specification and Drawing Sheets filed in connection with Application for Patent No 934/DEL/2000 dated 13.10.2000.

Witness my hand this 15th day of May 2001.

  
(B.P. Misra)

Joint Controller of Patents & Designs.

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FORM 1  
THE PATENTS ACT, 1970  
(39 of 1970)

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APPLICATION FOR GRANT OF PATENT  
[See Section 7]

We, INDIAN INSTITUTE OF TECHNOLOGY,, of Kanpur-208 016, Uttar Pradesh, India, hereby declare:-

- (a) that we are in possession of an invention titled "A MEGNETO-RESISTIVE  $\text{CrO}_2$  POLYMER COMPOSITE BLEND"
- (b) that the Complete Specification relating to this invention is filed with this application;
- (c) that there is no lawful ground of objection to the grant of a patent to us.

We further declare that the inventor(s) for the said invention is:-

SOLOMON SUNDAR MANOHARAN, an Indian citizen, of Department of Chemistry, Indian Institute of Technology, Kanpur-208 016, U. P., India,

We claim the priority from the application(s) filed in convention countries, particulars of which are as follows:

Not Applicable

We state that the said invention is an improvement in or modification of the invention, the particulars of which are as follows and of which we are the applicant:

Not Applicable

We state that the application is divided out of our application, the particulars of which are given below and pray that this application deemed to have been filed on \_\_\_\_\_ under section 16 of the Act.

Not Applicable

That we are the assignee or legal representative of the true and first inventor(s).

That our address for service in India is as follows:

LALL LAHIRI & SALHOTRA  
N-128, PANCHSHEEL PARK,  
NEW DELHI - 110 017,  
INDIA

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(011) 649-9923

Following declaration was given by the inventor(s) in the convention country:

ORIGINAL

I, SOLOMON SUNDAR MANOHARAN, an Indian citizen, of Department of Chemistry, Indian Institute of Technology, Kanpur - 208 016, U.P., India, referred to application as claiming to be the true and first inventor hereby declare that the applicant(s) who have signed this application is my assignee:

SOLOMON SUNDAR MANOHARAN

That to the best of our knowledge, information and belief the fact and matters stated herein are correct and that there is no lawful ground of objection to the grant of patent to us on this application.

Followings are the attachment with the application:

- (a) FORM 2 along with Complete Specification and Abstract (in triplicate),
- (b) Statement of Undertaking on FORM 3 (in duplicate),
- (c) Formal drawings (in triplicate);
- (d) Power of Attorney (will follow);
- (e) Fee of Rs. 5000/-

We request that a patent may be granted to us for the said invention.

Dated this 13th day of October, 2000.

*Ranjiv Mehta Dutt*  
OF LALL LAHIRI & SALHOTRA  
AGENT FOR THE APPLICANTS

To:

The Controller of Patents  
The Patent Office,  
at Delhi.



THE PATENTS ACT, 1970  
(39 of 1970)

093400  
13 OCT 2000

COMPLETE SPECIFICATION  
Section 10

ORIGINAL

"A MEGNETO-RESISTIVE  $\text{CrO}_2$  POLYMER COMPOSITE BLEND"

INDIAN INSTITUTE OF TECHNOLOGY,, of Kanpur - 208 016, Uttar Pradesh, India;

The following specification particularly describes the nature of this invention and the manner in which it is to be performed:-

The subject invention relates to a magneto-resistive  $\text{CrO}_2$  polymer composite blend for use in magnetic storage devices, such as in the audio and video tapes as magnetic read heads, magnetic field probes, or current voltage sensors in electrical devices and the process for the preparation of the same to provide a matrix for conducting and magnetic fillers to form a blend which in turn shows the desired magneto-resistive property.

The present invention also resides in the process for the preparation of a magneto-resistive polymer composite blend comprising a polymer, preferably Low Density Poly Ethylene (LDPE), the ferromagnetic filler preferably Chromium Oxide capable of exhibiting magnetic properties and an additive preferably conducting carbon brings about magneto-resistive properties in the composite.

The embodiment of the invention resides in the fabrication of the conducting polymer blend which would mimic the properties of ferromagnetism and metallic conductivity of the bulk  $\text{CrO}_2$  and to characterize its electrical property in the presence of an external magnetic field.

The object of the present invention is to improve the conductivity between the  $\text{CrO}_2$  grains embedded in a highly insulating polymer phase by adding an additive. The proportion of this additive in the polymer: $\text{CrO}_2$ :additive ratio varies from 3-10wt%. Percolation between the  $\text{CrO}_2$  grains is achieved even with 3% of this additive.

However a nominal composition of >3% is always preferred to keep the material below 100 Ohms. As a result of effective percolation between the magnetic  $\text{CrO}_2$  particles through a conducting additive network the response of the  $\text{CrO}_2$  -polymer blend can be measured by applying an external magnetic field.

Chromium (iv) oxide,  $\text{CrO}_2$  is known for its unique application as a tape material. It is a metal and at room temperature Ferro-magnet having a Curie Temperature of  $385^\circ\text{K}$  and at  $T=0\text{K}$ , it's a half metallic Ferro-magnet. This oxide is hydro-thermally prepared and has fine grained needles with acicular shape, a feature desired for magnetic storage applications. However, a new property such as magneto-resistance was realized in this compound recently. This property exhibits a large drop in resistance on application of an external field. The sensitivity with which it can change its resistance in the presence of an applied magnetic field determines whether this compound could be used as magnetic sensors.

Such magneto-resistive features have been exemplified in a variety of compounds which includes ferromagnetic Fe and Co based alloys, rare earth manganites. While, alloys exhibit this property in thin film configuration when stacked as magnetic and non-magnetic layers, manganites show such drop in resistance both in bulk and as thin films. This property if realized as a polymer, it can be exploited in a variety of applications due to ease of processing such a compound to any desired size and shape. This challenge therefore lies on the design of a polymer blend which can show such unique magneto-resistive behavior.

Electrically conducting polymer composite materials exhibiting positive temperature co-efficient of resistance effect have been in use in resistance switching devices for many years. These materials are characterized by a switch temperature at which the material resistivity changes by orders of magnitude. The most studied polymer composite system which exhibits this effect consists of polyethylene loaded with carbon black. At temperature below  $130^\circ\text{C}$  i.e. below the melting point of polyethylene there is an anomalous resistance which raises by orders of magnitude. This increase in resistance is believed to be due to the increased Carbon black particle separation which forms a dis-continuous polyethylene phase expansion. Upon melting, conducting polymer composite materials have been realized in

co-polymer blends too, such as polyethylene-polystyrene copolymers wherein conducting fillers such as carbon black can bring about electrical percolation at doping levels below 3wt%. As a result , a co-polymer which otherwise shows resistance of the order of M Ohms, shows below 100 Ohms when doped with conducting filler such as carbon black . This aspect in particular is used in sensor technology.

Conducting polymer composite materials consisting of a random distribution of a conducting filler throughout an insulating polymer are of interest for several applications.  $\text{CrO}_2$  being an important electronic material finds application as a recording material owing to its unique property of being ferromagnetic and metallic at room temperatures. Studies on  $\text{CrO}_2$  are limited owing to its thermal instability. The compound decomposes above  $300^\circ \text{C}$  which limits its application. The key tools in the design of a  $\text{CrO}_2$  based polymer blend are in appropriate polymer and selective localization of  $\text{CrO}_2$  grains in the blend. The best way to fabricate such a composite is to selectively disperse the  $\text{CrO}_2$  conducting chain with the filler in the insulating polymer matrix so that it forms a current conducting chain with the filler concentration as low as possible. An essential prerequisite for technological application is to retain the aciculate nature of the  $\text{CrO}_2$  grains during composite processing.

Accordingly, the present invention relates to a magneto-resistive  $\text{CrO}_2$  polymer composite blend for use in magnetic storage devices such as in the audio and video tapes as a magnetic read heads, magnetic field probes, or current voltage sensors in electrical devices comprising

- 88-93% by weight of the polymer preferably Low Density Poly Ethylene;

- 5-8% by weight of magnetic filler preferably  $\text{CrO}_2$  and

- 2-4% by weight of an additive.

The said polymer, preferably low density polyethylene is having a mesh size 200 micron and 99.9% purity and the said magnetic filler is in the form of powder.

The polymer composite blend is having the melting temperature of about 95° C .

The invention also resides in the process for the preparation of a magneto-resistive  $\text{CrO}_2$  polymer composite blend, comprising

- mixing the said polymer, said  $\text{CrO}_2$  and said additive as conducting carbon in a mortar pestle to obtain a homogenous mixture
- heating the said homogenous mixture at a temperature of 95-100° C to obtain a blended melt
- casting the said melt between two metal plates to obtain sheets by applying uniaxial pressure.

The pressure applied to obtain the said sheet is in the range of from 4-5.5Kpa.

Alternatively in the process for the preparation of a magneto-resistive polymer composite blend, the said polymer, said  $\text{CrO}_2$  and said additive were mixed to obtain a homogenous mixture, the said homogenous mixture is transferred into a die to obtain a pellet by applying a pressure of 4.5-5.5Kpa. The pellet so obtained is placed in a metallic mould and kept in a preheated hot press machine to allow the said polymer to melt at a pressure of 4 - 5.5Kpa and flow into thick sheet. The said mould is then cooled to obtain a film having magnetic properties.

The said mould used in the subject invention is preferably made up of aluminum and said hot press machine is uni-axial hot press machine

The size of the said films so produced varies from 10mm to 25mm in diameter depending on the size of the powder compact of the mixture.

The subject application may better be understood with reference to accompanying drawings. However, the same should not be construed to restrict the scope of the application as they are for illustrative purposes only.

### **BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWINGS**

Figure 1 depicts the X-ray diffractogram of the  $\text{CrO}_2$  polymer blend which shows magneto-resistive properties.

Figure 2 depicts the SEM micrograph of the  $\text{CrO}_2$ -polymer blend showing the features of grains. The black regions along the grains are predominantly carbon rich.

Figure 3 depicts the resistance versus temperature plot.

Figure 4(a-c) depicts the magneto -resistive property of the material at different temperature.

### **DETAILED DESCRIPTION OF THE INVENTION**

The invention describes a process in which a magneto-resistive material is embedded in a highly insulating polymer matrix and effective percolation between the  $\text{CrO}_2$  grains is achieved by adding another additive which has ability to bring about electrical percolation between the magneto-resistive material. The magneto-resistive material, in the presence of an external

magnetic field shows a unique drop in resistance of about 11% at 5K and at 5 Tesla. The nature of such magneto-resistance is exemplified in figures 4(a-c). The magneto-resistance ratio is defined as  $\Delta R/R_H$ .

The subject invention brings about a novel aspect of granular magneto-resistive system in which a polymer plays a role of a insulating matrix and the filler  $\text{CrO}_2$ , which is ferromagnetic and a metal behaves just as a bulk  $\text{CrO}_2$ , even with a composition of 10-20% by weight in 80-90% of the polymer.

The polymer used in the subject invention preferably is LDPE having commercial grade purity of 99.9% having a melting point of  $130^\circ\text{C}$ . To make a polymer blend, the physical mixture of the polymer,  $\text{CrO}_2$  and an additive is fed into a Brabender to make a melt at  $95-100^\circ\text{C}$ . The melt is then casted between two metal plates and drawn into sheets by applying a pressure of up to 5Kpa.

In an another embodiment, the polymer blend may be made by placing a powder compact of 5mm and thickness of 3mm in a metallic mould. The mould so used is preferably made up of aluminum. The metallic mould is then kept in a uniaxial hot press machine maintained previously at a set temperature of  $95^\circ\text{C}$ . The polymer melts and flows into the thick sheet, on applying the desired pressure. The pressure so applied is up to 5Kpa. The mould is kept at the desired pressure for about 5 minutes and then the pressure is allowed to release. The mould then is removed from the said hot press and is allowed to cool. The film so obtained in the polymer blend which is black, glossy and about 8-11 mm in dia.

The thickness and the size of the blend can be varied depending upon the die used, amount of material taken and the size of the pellet taken.

The weight percentage composition can be varied as follows :

Polymer	CrO <sub>2</sub>	Additive
90%	7%	3%
85%	11%	4%
80%	15%	5%
80%	12%	8%

The additive used is acetylene carbon which is a conducting form of carbon. The additive by itself does not show magneto-resistive property but performs only the act of providing percolation between magnetic grains. Percolation could be achieved with a minimum weight percentage of about 3%. Two probe resistance measurements show a resistance in 300K Ohms range for 3% additive. 5-8% additive was found to be conducive for the magneto-resistance measurements.

The mechanism of magneto resistance follows that the CrO<sub>2</sub> grains of the magneto resistive material tends to be fully spin polarized within each grain, meaning that the electrons of single grain have the same spin orientation. This polarization results from a ferromagnetic, electrons within individual grains have the same spin orientation, but the spin orientation of the electrons in adjacent grain will typically be different. In order words the transfer integral,

$$T_{ij} \approx \cos\theta_{ij}, \text{ where } i \text{ and } j \text{ are the grains aligned by an angle, } \theta.$$

The extent of intergrain electron transport in a material is highly dependent on the spin orientation of electrons in adjacent grains. It is relatively difficult for an electron of a certain spin orientation to move across the grains of a material when all the successive grains have electrons of different spin orientation. Upon application of the magnetic field to some spin polarized materials, at least some of the grains will arrange themselves such that their electrons can have the similar spins. The granular polarization makes electron transport across successive grains easier and thereby cause the



resistivity of the material to decrease. This relationship between applied magnetic field and resistance is magneto-resistance.

In the subject invention, the presence of insulating polymer as a major phase in the polymer blend acts as the insulating barrier providing magnetic decoupling between adjacent grains of the  $\text{CrO}_2$ . The insulating barrier however can be overcome with another additive such as Carbon which provides percolation between these grains. As a result a conducting thread is established. The desirable magneto-resistance offered by  $\text{CrO}_2$  grains is reflected hence by Fig.4a-c.

The subject invention employs the ability of the polymer blend to respond by way of resistive changes to small changes in applied magnetic field. Examples of article include sensor devices such as a magnetic read heads, magnetic field probes, or current voltage sensors in electrical devices.

Figure 1 shows the X-ray diffractogram of the  $\text{CrO}_2$  polymer blend. The first curve marked CB, shows the X-ray pattern of the additive alone. The curve marked 82468, denotes the  $\text{CrO}_2$ -polymer blend. The X-ray reflections correspond to both the additive and bulk  $\text{CrO}_2$ . The weakly intense peak with d values of 4.130 and 3.687 Angstrom denote the presence of the additive CB. The other reflections shown in figure 1 are due to  $\text{CrO}_2$ .

Figure 2 shows the scanning electron micro-graph of the as-prepared  $\text{CrO}_2$  polymer blend. The gray regions as shown in figure 2 are presumably the  $\text{CrO}_2$  grains and the dark regions interconnecting those grains are additive rich regions. Percolation between grains is achieved by doping up to 3-8% additive.

Figure 3 shows the plot of normalized resistance versus temperature plot for the  $\text{CrO}_2$  polymer blend with and without the application of an external magnetic field. Note the sharp decrease in resistance at low temperatures

below 10K . The drop in resistance arises out of inter-grain tunneling when two moments of neighboring grains are forced to align by applying an external magnetic field.

Figures 4(a-c) shows plot of magneto-resistance at different temperatures . The field dependence of resistance is more pronounced at temperatures below 4K and the percentage of magneto-resistance decreases with increasing temperatures. This is because the moments of each  $\text{CrO}_2$  grain in the polymer blend is so randomly oriented that the external field is unable to force the grains to orient parallel to each other . As a result there is no substantial change in the resistance. However at low temperatures, inter grain tunneling shows drastic change in resistance as a function of magnetic field.

### **EXAMPLES:**

#### **EXAMPLE 1:**

4% of the carbon black as additive and 16% of  $\text{CrO}_2$  and 80% of low density polyethylene were mixed together in a agate mortar pestle and the intimately mixed powder was compacted together as a pellet using a die of 5mm diameter. The powder was compacted to a thickness of about 4mm thickness by applying a pressure of about 3 Tonnes. The pellets so obtained were placed in a preheated die heated at  $95^\circ \text{C}$  . The die was closed and placed in a hydraulic press and a uni-axial pressure of about 5 Tonnes was applied . The polymer composite melts at the applied pressure and temperature to form a blend, which is circular in shape and having a thickness of about 1mm, forming the basis for preparing the  $\text{CrO}_2$  blend with varying composition. The thickness and shape of the blends can be changed with the configuration of the die that is employed.

#### **EXAMPLE 2:**

5% of the carbon black as additive and 14% of  $\text{CrO}_2$  and 81% of low density polyethylene were mixed together in a agate mortal and the intimately mixed powder was compacted together as a pellet using a die of 5mm diameter. The

powder was compacted to a thickness of about 4mm thickness by applying a pressure of about 3 Tonnes. The pellets so obtained were placed in a preheated die heated at  $95^{\circ}\text{C}$ . The die was closed and placed in a hydraulic press and a uni-axial pressure of about 5 Tonnes was applied. The polymer composite melts at the applied pressure and temperature to form a blend, which is circular in shape and having a thickness of about 1mm, forming the basis for preparing the  $\text{CrO}_2$  blend with varying composition. The thickness and shape of the blends can be changed with the configuration of the die that is employed.

A rectangular portion of the blend was employed for electrical resistivity studies. Four probe contacts were made with electrolytic grade silver paint and insulated copper wire. A He Cryostat, Oxford instrument was employed for studying the resistance versus temperature plot. The samples were cooled at 4.2K and the resistance as a function of applied magnetic field 0 to 5 Tesla was measured at different temperatures.

The subject application is a mere statement of invention, where various modifications and alterations are possible without deviating from the scope of the invention, hence the present application should not be construed to restrict the scope of the invention.

WE CLAIM:

1 A magneto-resistive  $\text{CrO}_2$  polymer composite blend for use in magnetic storage devices such as in the audio and video tapes as magnetic read heads, magnetic field probes, or current voltage sensors in electrical devices comprising

- 88-93% w/w of the polymer preferably Low Density Poly Ethylene;
- 5-8% w/w of magnetic filler preferably  $\text{CrO}_2$  and
- 2-4% w/w of an additive.

2 The polymer composite blend as claimed in claim 1, wherein the said additive is conducting carbon.

3 The polymer composite blend as claimed in claim 1, wherein the said polymer is in the form of powder.

4 The polymer composite blend as claimed in claim 1, wherein the said magnetic filler is in the form of powder.

5 The polymer composite blend as claimed in claim 1, wherein the said additive is in the form of powder.

6 The polymer composite blend as claimed in claim 1, wherein the said low density polyethylene is having a mesh size 200 micron and 99.9% purity.

7 The polymer composite blend as claimed in claim 1, wherein the melting temperature of the said polymer is  $95^\circ\text{C}$  under a pressure of 5Kpa.

8 A process for the preparation of said magneto-resistive  $\text{CrO}_2$  polymer composite blend as claimed in claim 1, comprising

- mixing the said polymer, said  $\text{CrO}_2$  and said additive in a mortar pestle to obtain a homogenous mixture

- heating the said homogenous mixture at a temperature of 95-100°C to obtain a blended melt
- casting the said melt between two metal plates to obtain sheets by applying the pressure.

9 The process as claimed in claim 8, wherein the said pressure is applied to the metal plates to obtain said sheet is from 4 - 5.5 KPa.

10 A process for the preparation of a magneto-resistive  $\text{CrO}_2$  polymer composite blend as claimed in claim 1, comprising , mixing the said polymer, said  $\text{CrO}_2$  and said additive as conducting carbon to obtain a homogenous mixture , transferring the said homogenous mixture in a die at a pressure of 4.5-5.5 KPa to obtain a pellet , placing the said pellet in a metallic mould and keeping the said mould in a preheated hot press machine maintained at a temperature of 95°C at a pressure of 4-5.5 KPa, allowing the polymer to melt and flow into thick sheet, cooling the said mould to obtain a film having magnetic properties .

11 The process as claimed in claim 10, wherein the said mould is made up of metal , preferably of aluminum.

12 The process as claimed in claim 10, wherein the diameter of the said films varies from 10mm to 25mm depending on the size of the powder compact of the mixture.

13 The process as claimed in claim 10, wherein the said hot press machine is uni-axial hot press machine

14 A process for the preparation of magneto-resistive  $\text{CrO}_2$  polymer composite blend substantially as herein before described with reference to the foregoing examples.

15 A magneto-resistive  $\text{CrO}_2$  polymer composite blend substantially as herein before described with reference to the accompanying drawings and foregoing examples.

DATED THIS THE 13TH DAY OF OCTOBER, 2000

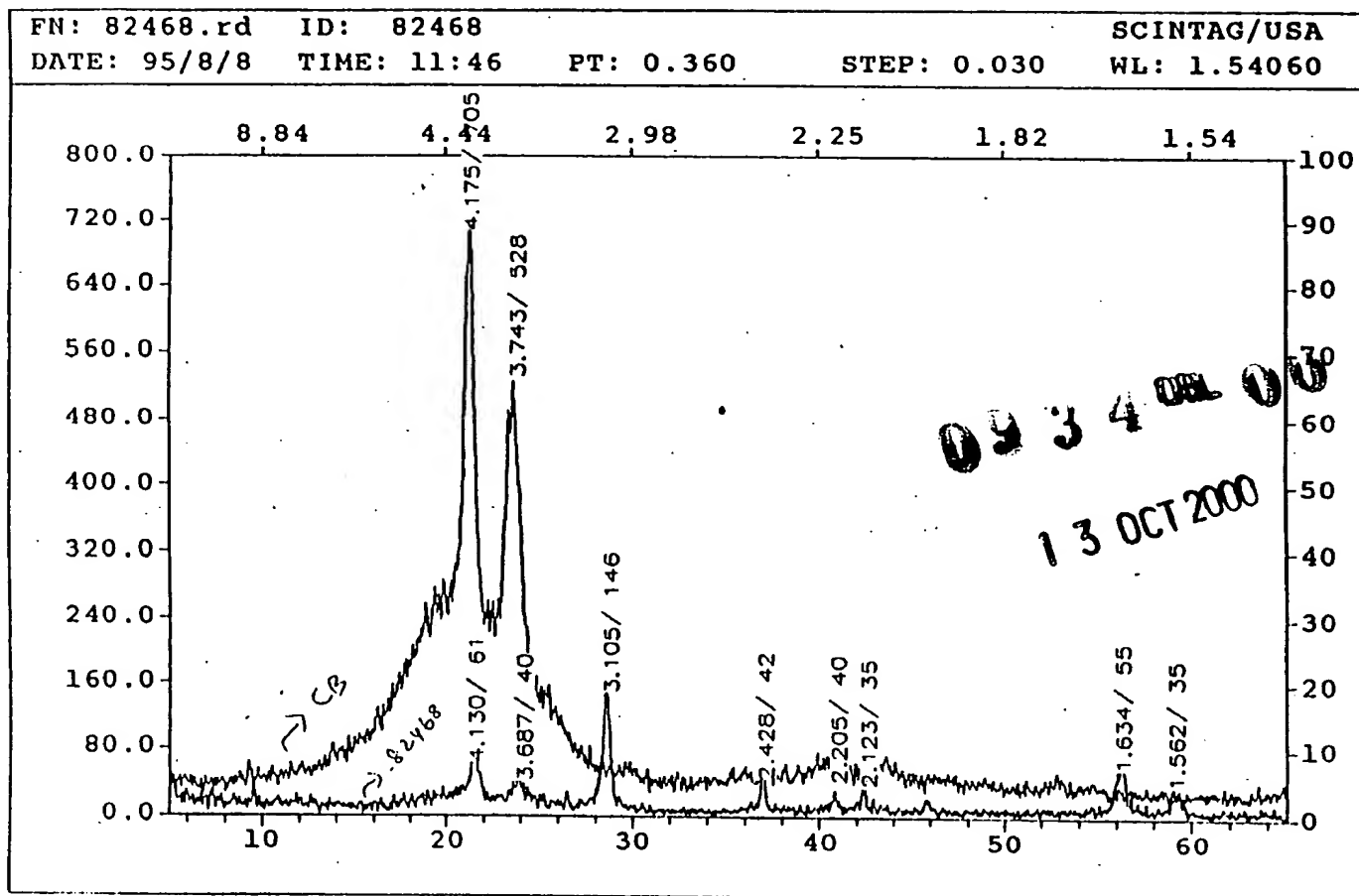
*Ranjiv Mehta Dutt*  
LALL LAHIRI & SALHOTRA  
ATTORNEYS FOR THE APPLICANTS

## ABSTRACT

A magneto-resistive  $\text{CrO}_2$  polymer composite blend comprising 90-95% by weight of the polymer preferably Low Density Poly Ethylene, 5-10% by weight of magnetic filler preferably  $\text{CrO}_2$  and 2-4% by weight of an additive, prepared by the process of mixing the said polymer, said  $\text{CrO}_2$  and said additive in a mortar pestle to obtain a homogenous mixture, the said mixture is heated at a temperature of 95-100°C to obtain a blended melt, the said blended melt is then casted between two metal plates to obtain sheets by applying the pressure.





FIG. 1

Ranjiv Mehta Dutt  
of Lall Lahiri & Salhotra  
Agent for the Applicants.

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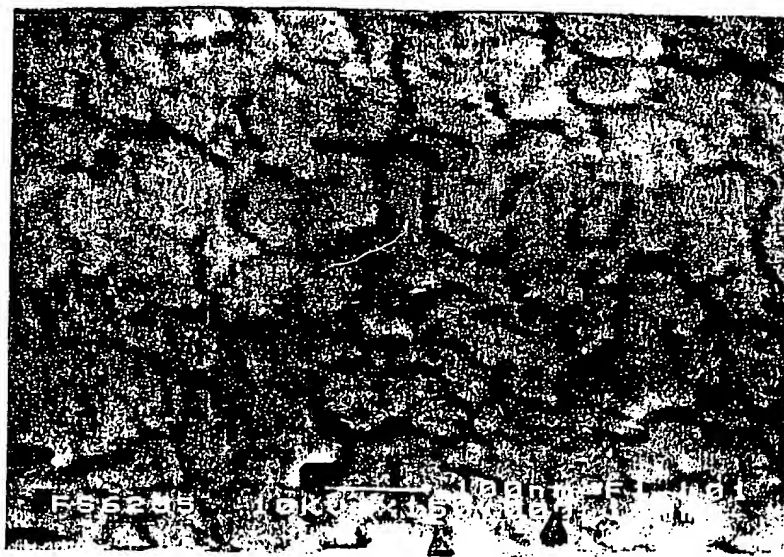
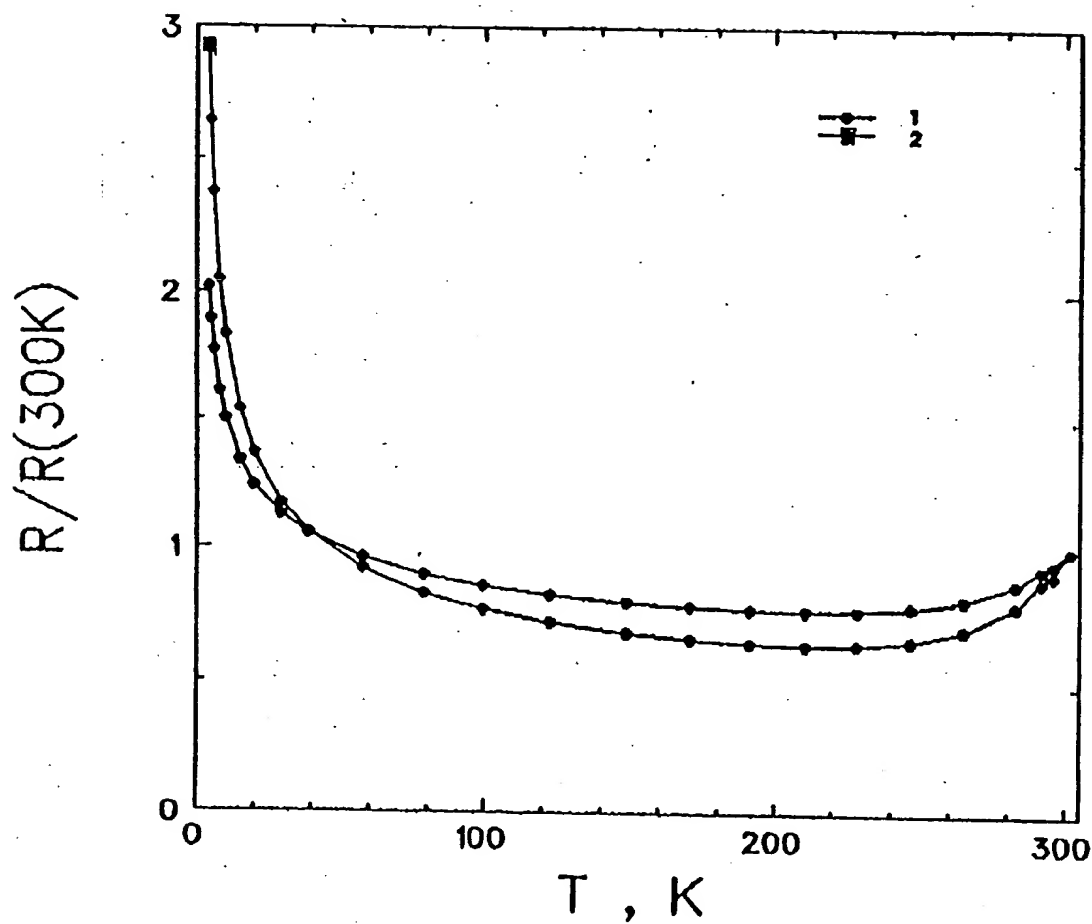


FIG. 2

Ranvir Mehta Dutt  
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FIG. 3

*Ranin Mehta*  
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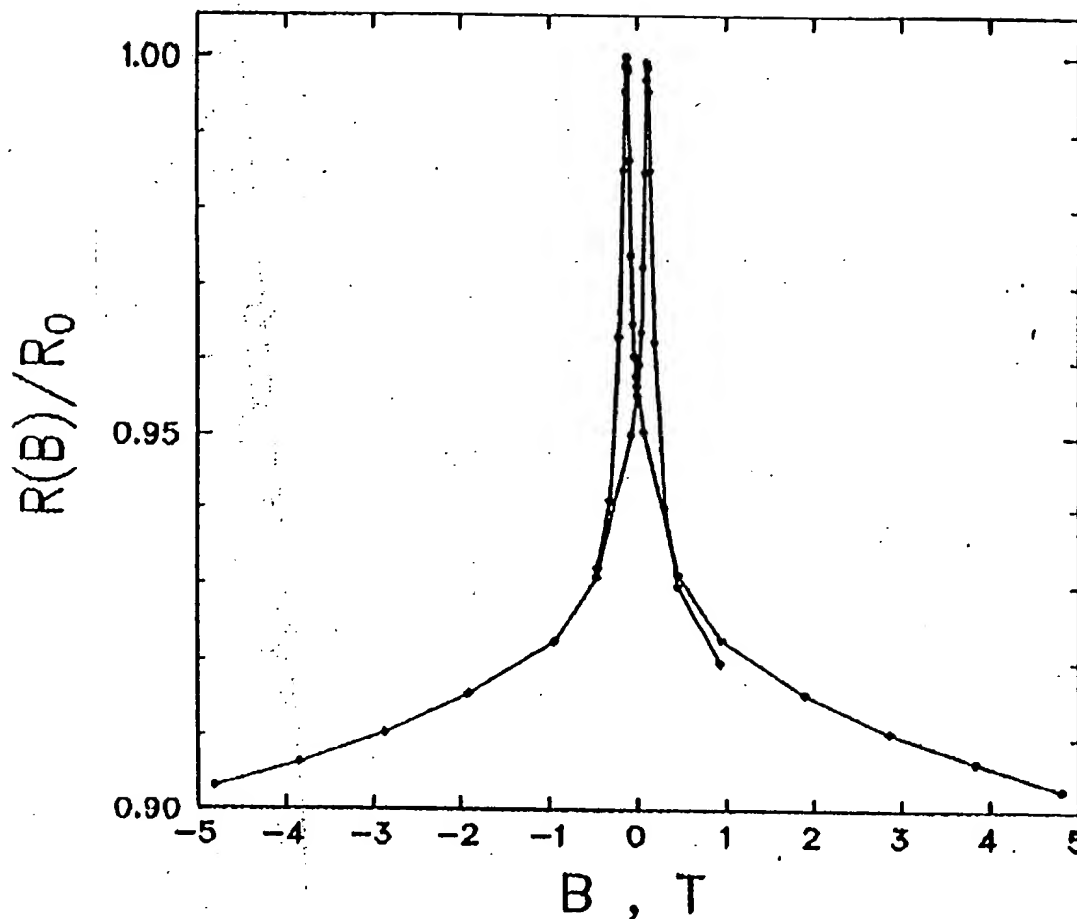


FIG. 4(a)

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*Ranjna Kanta Dutt*  
of Lalit Lahiri & Sahotra  
Agent for the Applicants.

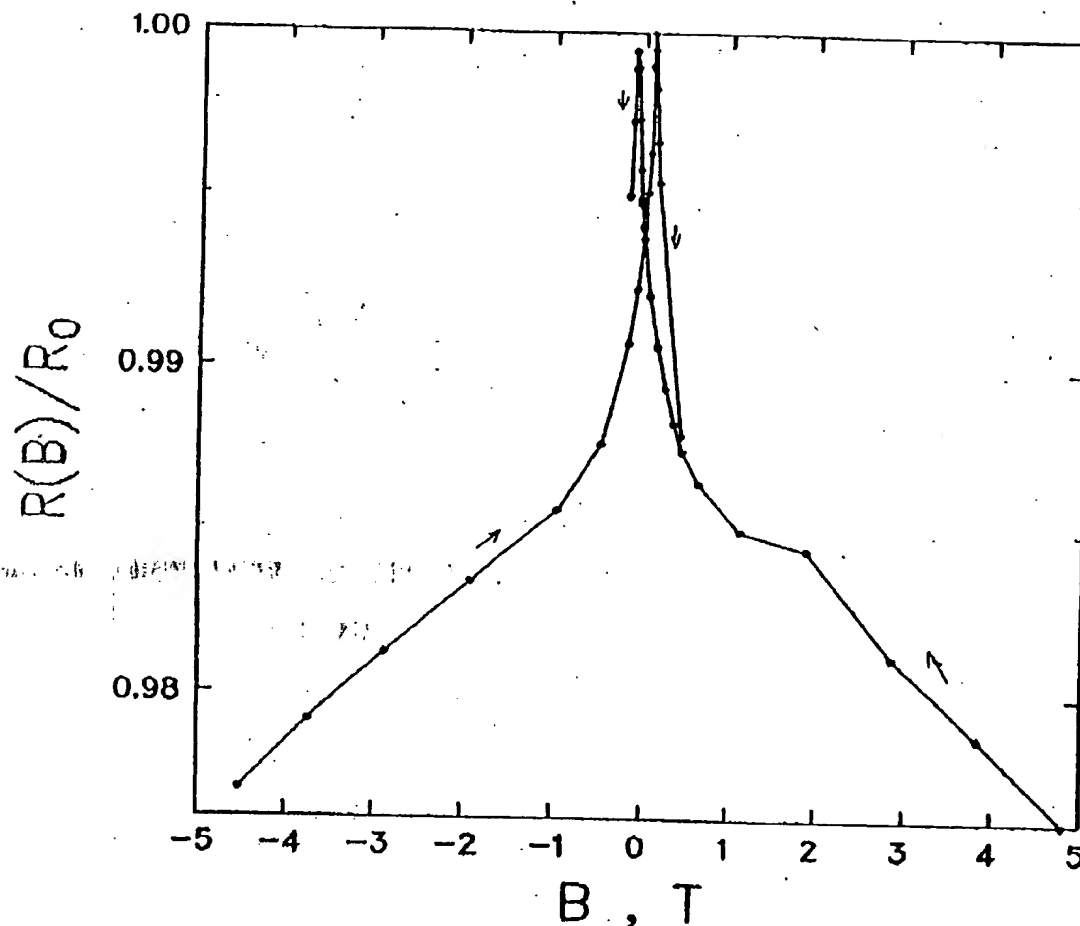


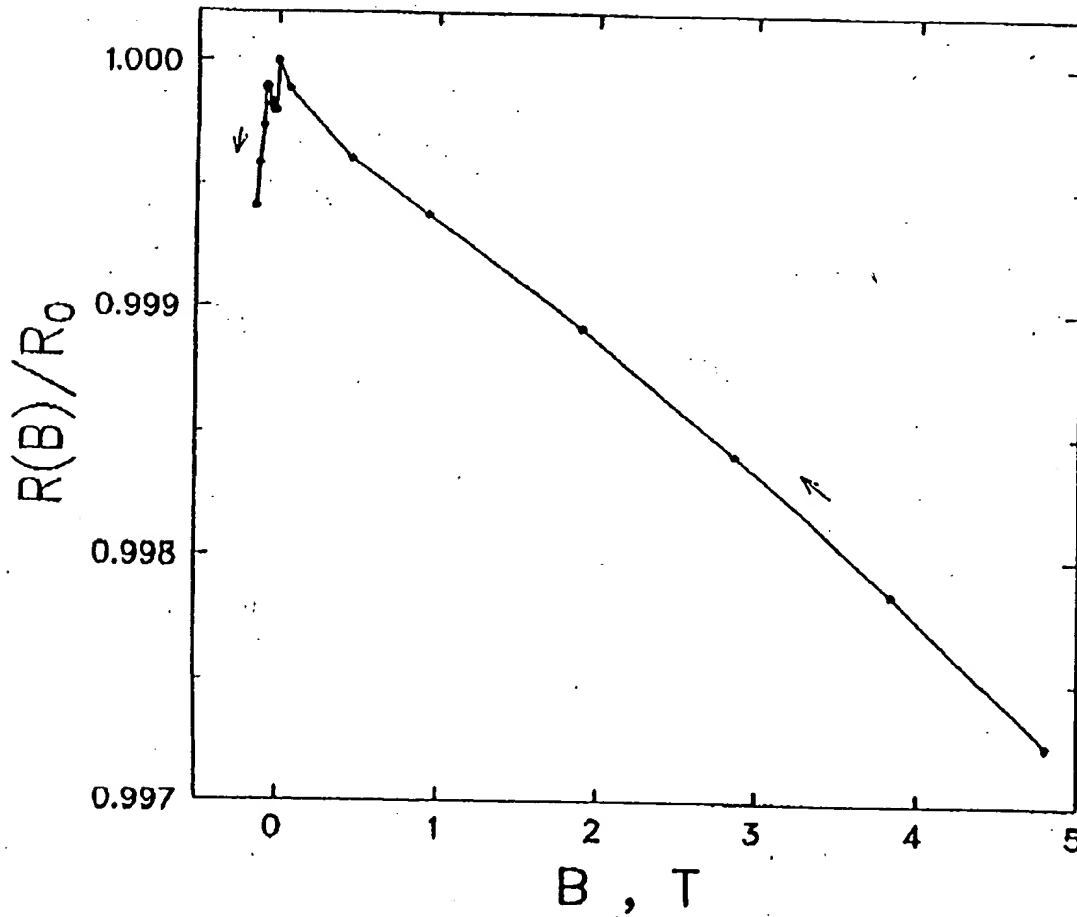
FIG. 4(b)

13 OCT 2000

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Ranjeet Mehta Dutt  
of Lali Lehari & Associates  
Agent for the Applicant.

ORIGINAL



ORIGINAL

FIG. 4(c)

3 OCT 2000

09 34 10.0

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